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MECHANICAL, THERMAL AND TRIBOLOGICAL CHARACTERIZATION OF PURE AL FUNCTIONALLY GRADED MATERIAL IN SITU WITH ALUMINA, COPPER AND ZINC STERATE AS LUBRICATING AGENT

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ABSTRACT

The technological advancement in the sector of materials science is increasing in a rapid and futuristic manner. This, in turn is enormously affecting the manufacturing sector of different components. Today, every industry is striving very hard to satisfy the needs and demands of the customer and adopting very strict design to inspection procedures. Therefore, it is very essential to develop the materials which not only gives a boost to the manufacturing sector, but also satisfies the customer needs and requirements. There is a lot of research done in the field of material science from alloys to the composites but they have their own advantages and limitations. Today, there is lot of research done in the domain of functionally graded materials, which not only produce light weight materials, high strength to weight ratio, but also possesses superior qualities than metal matrix composites, in terms of material composition and functional requirements [2]. So, in this regard, keeping in view of the automobile sector, this paper is keen in focusing of developing functionally graded material, considering pure Al as the matrix constituent and alumina (Al_2O_3), copper(Cu) as the reinforcing constituents and zinc sterate as lubricating agent. The uniform mixing of the constituents are done by adopting rule of mixtures, and optimum mix ratio is processed and blended with the aid of ball milling machine specified with ASTM E 23 standards [4]. The developed constituents are then compacted using compacting machine with definite compaction and ejection process and the compaction load is varying in between 16.8KN to 18 KN and ejection load varies between 10.5 KN to 12KN.The synthesized green compacts are then sintered at 1100°c for about 4 hours and remained till they are furnace cooled. Then, the processed dry compacts are being characterized for their mechanical properties such as the compression strength and hardness. Micro structural characterization is conducted with the aid of scanning electron microscopy (SEM), X-rays diffraction(XRD)&energy dispersive X-rays analysis(EDX), and tribological characteristics such as wear rate, coefficient of friction and frictional force are successfully accessed by the DUCOM pin on disc wear test apparatus TR-201 with 68 HRC and various graphs are drawn to determine various characteristics of the material with the functional parameters such as sliding velocity, wear time and wear distance in wear characterization.

Original Article

The salient points inferred is, while mechanical characterization, as the amount of alumina is increased while keeping the cu weight % fixed, the compressive strength of the material is linearly decreased and then at a sudden rate. The compression strength is increased linearly from inner region to the outer region with increase in weight percentage of Cu. The micro structural analysis also inferred that, there is a valid inclusion of alumina and copper particles with the matrix constituents having a strong bonding with reference to various layers of the region.

KEYWORDS: Composition, Blending, FGM, Sintering, Weight Percentage & Strength

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1. INTRODUCTION

The concept of functionally graded materials (FGM) was developed in Japan, in the year 1988, founded by functionally graded materials forum, to manufacture aeronautical devices which will behave as thermal barriers for the bulk material to withstand higher temperatures of the order 10,000–20,000 degrees Celsius. Later on, they had been modified into several forms, several stages, several parameters and different operating conditions to fulfill different sectors such as defense, aeronautical, civil and mechanical structures, manufacturing and automobile industries.

The main reason why there is lot of research activity in the field of functionally graded materials is that they possess a unique and special property as varying in material constituents as the variation of volume fraction of the material occurs, this predominant quality makes FGM a special material and stands apart from the conventional materials. There is not only a vast research being conducted in FGM, but also there is lot of futuristic scope in FGM because as the research is being extended, new and unknown facts are being derived out from these functionally graded materials. In the forthcoming days, it will nevertheless be that the conventional materials will be replaced by these FGM's. In engineering materials, the FGM's are mainly evolved to replace the alloys and composite, which have their own limitations such as regarding the alloys, the material is heavy and needs lot of stipulated time and procedure to manufacture and produce the desired output within the cumbersome time period and also the catastrophic failure rate is high [5]. Regarding composite materials, even though they are light weight, high strength to weight ratio, high stiffness. The processing tools and techniques are not standardized and also the failures with respect to cyclic loading are high and are only limited to very few industries and applications.

The manufacturing of FGM is very important task, with which, the reliability of the end product is assessed. The FGM are manufactured mainly with the processing techniques such as solid free form, centrifugal stirring and powder metallurgy to synthesize bulk FGM, but whereas vapour deposition, plasma spraying and ion beam assisted deposition to produce thin film FGM. Although the techniques are very low in number, but these procedures are standardized and specified and they meet the production needs and customer requirements. At present, the FGM are manufactured adopting G 39 ASTM standardization.

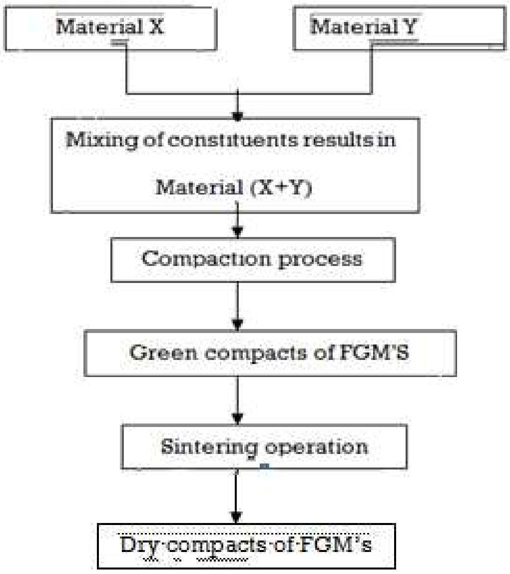


Figure 1: Showing the Block Diagram of Processing of Functionally Graded Materials.

2. LITERATURE REVIEW

N. Radhika et.al in her research titled, development of functionally graded aluminium composites using centrifugal casting and influence of mechanical and wear properties, have produced with the aid of centrifugal casting of the constituents pure aluminium as the base metal boron carbide, silicon carbide, alumina and titanium boride and inferred that the outer peripheries of the FGM exhibits higher hardness except in AlB4C and the outer zones of all the composites exhibits higher tensile strength.

Dr. C.N. Chandrappa et.al [4] in his research titled, synthesis and characterization of Al-SiC functionally graded metal matrix composites using powder metallurgy techniques, has synthesized FGM's using conventional powder metallurgy method observed that high volume fraction of SiC caused clustering of carbide phase at grain boundaries, which restricts inter particle contacts further becomes a wall for densification, and he has also observed that optimum results are obtained at weight percentage of 25% and 320 mesh size of SiC particles inclusion.

Zhang Zhong tao et.al [1] in his research titled, Preparation of Al/Si functionally graded materials using ultra soic separation method, has successfully synthesized the standardized Al-Sic FGM and also determined that as the percentage composition of SiC increases the FGM becomes very hard and making the material brittle, but the higher hardness and compressive strength is achieved with 7.5% of SiC composition.

Kushagra gupta et.al [7] in his research titled, synthesis of functionally graded material using powder metallurgy, has successfully synthesized Aluminium A7075 with titanium diboride (TiB2) and also his findings said that self lubricating are developed in FGM when the porosity level is increased by 20% in FGM plate, the hardness of the FGM is enormously increased with the increase of TiB2 and increase in the sintering time enhances the hardness, and also observed a successful phase change in metallurgical characterization.

Amir Arifin et.al in his research paper, material processing of hydroxyapatite and titanium alloy composites as implant materials using powder metallurgy, processed successfully HA/Ti FGM's successfully and also inferred that as the

percentage of titanium is increased, the hardness and the compaction capability of the FGM increases, and also he has observed perfect bonding within the elements and minimal voids and wear rate is also deceased at considerable rate.

Madhusudan et.al in their research paper titled, effect of mould wall thickness on rate of solidification of centrifugal casting, successfully formulated procedure to determine the optimal thickness of the mould material in the production of FGM in centrifugal casting and also inferred various parameters effecting the FGM production starting from preheating, molten metal heating and solidification rate.

Sivaprasad katakam et.al in his research on microwave processing of functionally graded material, has successful standardized the microwave method in FGM synthesis. He not only determined the optimal method, but also inferred various step by step procedures in microwave method in FGM syntheziation and characterization; he also inferred that the mixing of materials is to be adopted with Thompson method for the mixing of materials, and also discussed thoroughly various techniques and devices in the micro structural and mechanical characterization.

Ravindrababu et.al in his research paper titled, design, development and fabrication of AL functionally graded metal matrix composites, has successfully synthesized various particulate reinforcements and also inferred that the layer by layer deposition of the material crucially effects the compaction time and sintering temperature.

3. EXPERIMENTAL PROCEDURE

3.1 Materials

Matrix constituent: Pure aluminum of purity 98% and particle size 100 microns are taken in powdered form.

The main reason to choose pure aluminium as the matrix material is because, most of the growth sectors are utilizing aluminium as the major constituent either in the form of alloying material or particulate or grain form. Pure aluminium is not only used in industrial sector, but also we use in our daily life in one form or the other, and also it has enormous applications. It is not only a low density material available in the nature but it is availed easily, processed and manufactured to different components in various fields. When this material is mixed with other materials, the composite formed thus after that is superior to that of the parent material and behave excellently in tough and hazardous situations.

Table 1: Exhibiting the Composition of Mass Fractions in Percentages of Different Metals with Respect to the Aluminium Metal

Si	Fe	Mn	Mg	Ti	Al
0.46	0.18	0.026	0.45	0.021	98.021

Reinforcements

3.1.1 Alumina (Al_2O_3)

Alumina is called by its chemical name aluminium oxide is taken as first reinforcing agent as it possess various number of applications in industrial to house hold purposes. It is not only low density constituent but also easily availed and processed at low cost. It serves its purpose and possess superior qualities when combined with the parent material such as Al, Fe, Cu, Zn and Nickel resulting in superior material which is served in present day industries. In our research we preferred to have alumina in fine powdered form of 20 microns grain size

Table 2: Chemical Composition of Alumina having Different Constituents in terms of % Mass Fractions

Al_2O_3	Na2O	Sio2	Fe2O3
99.2	0.4	0.1	0.3

3.1.2. Copper(Cu)

Research and application towards the copper metal is very limited, and mostly it is used extensively in electrical equipment, as it is an excellent conductor of electricity having coefficient of thermal conductivity as 385 W/m-K. But, the research towards copper as the ingredient in material science and its applications is limited, so in order to overcome all those limitations in our research paper, we have emphasized to consider Cu in fine powdered form as the second reinforcing element and investigate its effect on the materials after the combination.

Table 3: Shows the Chemical Composition of Copper having Different Constituents in terms of % Mass Fractions

Ni	Pb	Sn	Sb	Cu
4.79	1.11	0.654	1.15	92.1

3.2 Experimental Equipment and Methodology

The matrix and the constituents are to be very carefully handled as they are taken in the powdered form and should be kept in jar with air locked such that, there is no chemical reaction with the atmospheric air and contaminants before and after the experimentation. The powders are weighed with the help of micro weighing machine of ASTM D6051-15 standards by wearing hand gloves according to the procedure adopted in mixing of constituents in different methodologies [9]

After dividing the matrix and the constituents as per the calculations of mass densities, total volume of the FGM is to be maintained constantly throughout the experimentation. The separated powders are now ready for the ball milling process.

Table 4

Layer Number	Matrix Constituent (%)	Reinforcing Constituents (%)	
		Alumina	Copper
1	100	Nil	Nil
2	95	5	Nil
3	90	5	5
4	85	5	10
5	80	10	10

Table 4 shows the chemical composition of the layer by layer size of the FGM with matrix and the reinforcements in terms of % mass fractions

In order to have uniform blending between the matrix and the reinforcing constituents, we have utilized the ball milling machine, which not only provide uniform mixing of the powders but also at different speeds and radius of rotation . The powders, which are ball milled after then bonding with the other layer constituents when compaction performed without ball milling and the characterization of such composites are showing considerable results rather than products synthesized without ball milling operation.

3.2.1 Compacting Machine

The materials after the ball milling are ready for the uniform, controlled and automatic compacting machine as per ASTM G47 standardization. The superior quality when compared to the ordinary compacting machine is that, we can simulate the compacting operation without the specimen and could rectify the errors before loading of the operation, and the machine also calculates the optimal and exact compaction and ejection force, and it is very much essential to have uniform compaction process as this process decides the sustainability of the prepared FGM’s.

(a) (b) (c)

Figure 2: (a) Represents the Compaction Process in the Compacted Die (b) Represents the Ejection Process in the Ejection Die (c) Represents the Compaction and Ejection Dies with Stoppers.

The compaction process is so uniform and perfect that we can see the process through the system and non uniformity of the process can be stopped at any stage of the operation and the errors are rectified, and can continue the operation although the compaction process is flawless. The compaction die is taken and the powders are poured manually through the hopper, and automatic rammer is provided to have uniform ramming of the blended powders (100%Al) to the bottom most layer and subsequently the next layer consists of (95%Al,5%Al_2o_3) is placed through hopper and uniformly rammed up to the top most layer consists of (80%Al,10%Al_2O_3,10%Cu) [10]. Now, the compaction die consists of layer by layer deposition of matrix and reinforcements, in terms of their mass fractions, now it is carefully placed under the punch and it is compacted with a force of 16.8KN for three specimens and 17.4KN for remaining three specimens, because it gives the uniform compaction of green compacts. Proper care should be taken that the green compacts need not to be removed from the compaction die forcibly. The punch is retrieved and the compaction die is reversed and below of it, ejection die is placed carefully without any eccentricity. Now, the punch is moved carefully and it is made to move until the specimen comes out through the ejection block freely. If the compaction is non-uniform, the specimens don't come out from the ejection die, or it may fail or cracks appear on the green compacts. In our research, a total of 12 specimens are produced to quantify different characterizations.

3.2.2. Sintering Operation

The processed green compacts are then prepared for sintering operation, with the aid of tubular furnace of Argon gas entrapment. Before sintering, the specimens are preheated at a temperature of 300 degrees centigrade, in order to easily avail the heating of the tubular furnace rather than heating at the room temperature. A batch of 3 specimens are taken at once and placed in air tight box and inserted into the cylindrical section of the furnace, and the entry of the furnace is closed with the help of fasteners. The sintering temperature and time is considered as per the researchers from their adopted methodologies and optimal parameters. Sintering temperature is considered to be 1100°C and the sintering time to be 240 minutes [11]. The knobs are rotated as per the speculated time and temperature and the argon gas impingement is provided so as to avoid contamination with the atmospheric air and unnecessary reactions. After attainment of highest temperature, the specimens are left in the furnace to cool slowly and gradually, as the rapid cooling can affect the mechanical and micro structural characterization. Further, the FGM's are left entirely in the furnace for 24 hours to cool at a very slower and natural rate. The same process is repeated for all the 4 batches, each consisting of 3 specimens.

Figure 3: Shows the Tubular Furnace with Argon Gas Attachment for Sintering Operation.

Figure 4: Represents the Specimens Processed after Sintering Operation in Tubular Furnace.

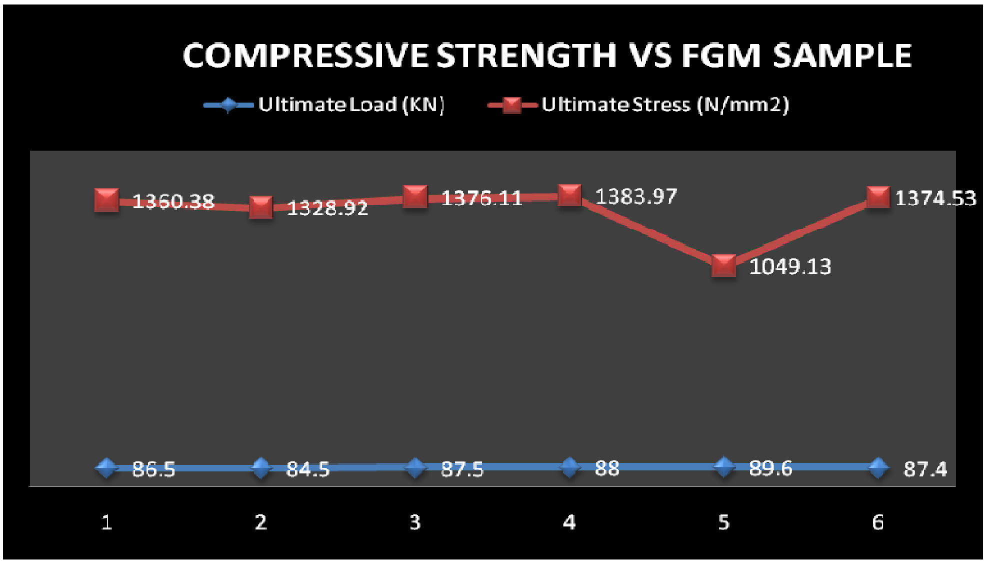
4. RESULTS AND DISCUSSIONS

4.1 Compression Strength

The compression test, a perfect test to determine the strength in the material when the cross section of the material is very low was conducted in our analysis. The specimen produced possesses the length as 13 mm and diameter as 9 mm, making the cross sectional area to be 63.585 mm2, so the tensile strength is its limitation, therefore compressive strength is preferred. In this procedure, the specimens are placed between two jaws in which, one is fixed and the other jaw is movable. Universal testing machine is taken as equipment in assessing this character, as the load is applied across the entire surface area of two opposite faces and the plates are pushed together to make the sample to flatten [13]. A compressed sample is usually shortened in the direction of the applied forces and expands in the direction perpendicular to the force. A compression test is essentially the opposite of the more common tension test.

Table 5: Shows the Chemical Composition of Copper having different Constituents in terms of % Mass Fractions

Sample No	Sample Name	Ultimate Load (KN)	Ultimate Stress (N/mm^2)
1	Al –Al_2o_3-Cu	86.5	1360.38
2		84.5	1328.92
3		87.5	1376.11
4		88	1383.97
5		89.6	1049.13
6		87.4	1374.53



Graph 1: Represents the Variation of the Compression Strength with Respect to the Specimen.

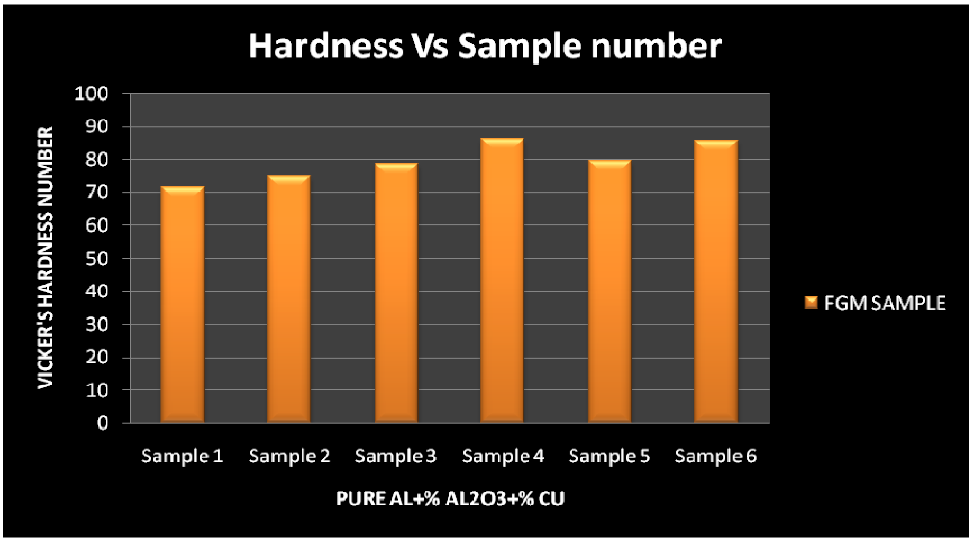
In the above graph, we can clearly observe that the compressive strength of all the specimens is not constant, but varies uniformly at a lower rate but almost constant ranging from a mean value of 86 KN, which is a high factor resisting the compressive loading, but when we consider the conventional aluminium alloys, they fail at a value of 58 KN which show the superiority of the FGM when compared to conventional materials, but due to the limitation in the die design, we could only make a cross sectional area of 63.585 mm^2 otherwise we could manufacture a component having 8000 mm^2 cross sectional area, and thus mounting the compressive strength to be more than 300KN. The reason that it shows high compressive strength is due to the uniform layer by layer deposition of aluminium with alumina and copper particles, resulting in the strong bonding between the constituents and minimal number of voids, and is also one of the predominant factors, which mainly effects the compressive strength.

4.2. Hardness Measurement

In order to assess the characteristic which resembles the resistance to indentation, hardness is the measurable sign. The hardness has become the vital parameter to assess the material that withstand higher loading without causing any imperfections on the material, and also to avoid catastrophic failure, hardness is judged as the dominant parameter. So in this regard, the FGM's are tested with the help of vicker's hardness tester for its micro hardness due to cross sectional constraint. The specimens are kept in the tester and the diamond indentor is placed uniformly and adjusted to suitable space for nil imperfections and dent is marked, and it is then measured by using metallurgical microscope and the procedure is repeated for all the specimens.

Table 6: Represents the Variation of the Hardness of the FGM with Respect to Different Specimens

Sample No	Sample name	Trail-1	Trail-2	Trail-3	Hardness
1.	Pure Al -Alumina -Copper FGM	1.7	1.6	1.8	71.587
2.		1.7	1.7	1.6	75.001
3.		1.6	1.7	1.6	78.62
4.		1.6	1.6	1.5	86.53
5		1.6	1.5	1.7	79.54
6		1.5	1.6	1.6	85.68



Graph 2: Represents the Variation of the Hardness with Respect to the Specimen.

4.3. Thermal Conductivity Test

Researchers have done enormous work in the field of materials to calculate the effective thermal conductivity of composite material. Also, models are developed for the two phase mixture.

Maxwell in 1873 had designed a model to determine the distribution of molecules in composites, and he had assumed the molecules are randomly distributed and having spherical shape which are non-interacting homogeneous and dispersed in homogeneous medium. The expression is shown below

$$k_c = k_m \left[\frac{k_p+2k_m+2\emptyset(k_p-k_m)}{k_p+2k_m-\emptyset(k_p-k_m)}\right] \quad (1)$$

So many articles are written, keeping in view of the material that describes the models for the estimation of effective thermal conductivity of a two-component composite. In the basic model, the effective thermal conductivity is calculated by considering the material in which, the molecules are arranged either in series or parallel with respect to the direction of heat flow. For these arrangements fundamental correlation has been given.

Series heat conduction model:

$$\frac{1}{k_c} = \frac{1-\emptyset}{k_m} + \frac{1}{k_p} \quad (2)$$

Parallel heat conduction model

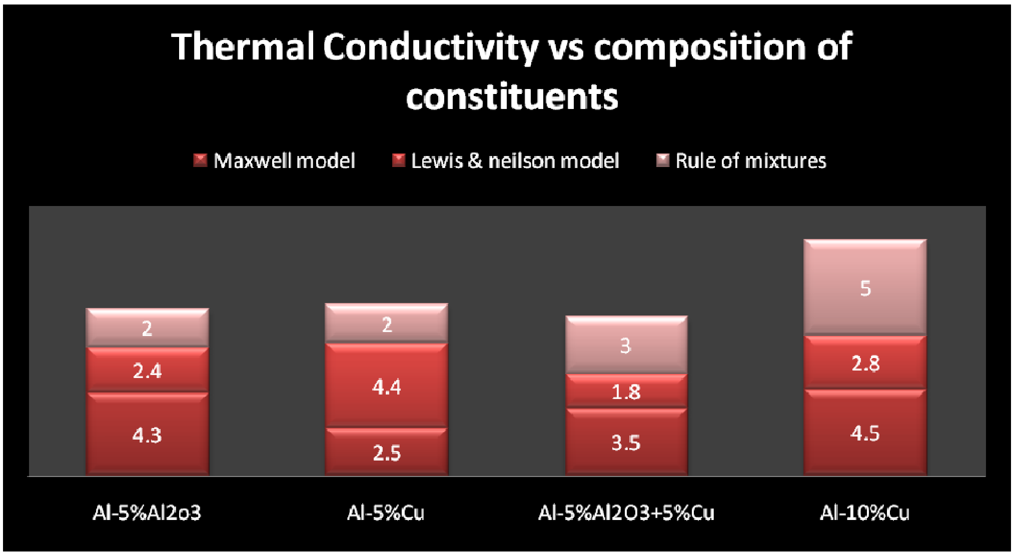
$$k_c = (1-\emptyset)k_m + \emptyset k_p \quad (3)$$

Where k_m, k_p are the thermal conductivities of the matrix and the particulate respectively, k_c is the effective thermal conductivity of the composite and $\emptyset$is the volume fraction of theparticulate. Equations (1), (2) and (3) are representing the thermal conductivity behavior of the materials by the assumptions of Maxwell, Lewis and rule of mixtures methodology.

In our research paper, we have given emphasis on determining the thermal conductivity based on three methodologies i.e. Maxwell model, Lewis & Neilson model and rule of mixtures.

Table 7: Shows the Variation of Thermal Conductivity with Variation in the Reinforcements and Criteria Model.

Criteria	5% Al2o3	5%Cu	5% Al2o3+5%Cu
Maxwell model	232.34	297.39	380.36
Lewis & Nielson model	233.88	317.72	471.32
Rule of Mixtures	182.21	182.21	182.21



Graph 3: Represents the Variation of the Thermal Conductivity of FGM with Respect to the Composition of Constituents and Thermal Model.

From these above thermal conductivity graphs, we infer that the variation of thermal conductivity when the reinforcement constituent alumina is considered, the variation is of low amount when all the three criterion models are considered, and the highest is achieved in Al-10%Cu of 4.5 W/m-K and lowest is 2.5W/m-K at 5% alumina reinforcement considering Maxwell model. While the Lewis & Neilson model is having considerable downfall in the thermal conductivity values, the highest being 4.4 W/m-K at 5% copper and the lowest magnitude is 1.8 W/m-K at equal proportion of 5% alumina and copper, but when we consider the rule of mixtures as the criterion, the increase is slow, but it has considerable effect in the thermal behavior

4.4. Micro Structural Investigation

Figure 5.8 shows the Elemental wise analysis of the FGM reinforced with different weight fractions of Cu which was conducted in order to assess the bonding with respect to the parent material and also to investigate the chemical composition of the reinforcing agents with the base metal and also the phase change in the grain boundaries. The EDX confirms the presence of Al and Al_2O_3, copper and some minor impurities such as SiO_2 and Mgo.

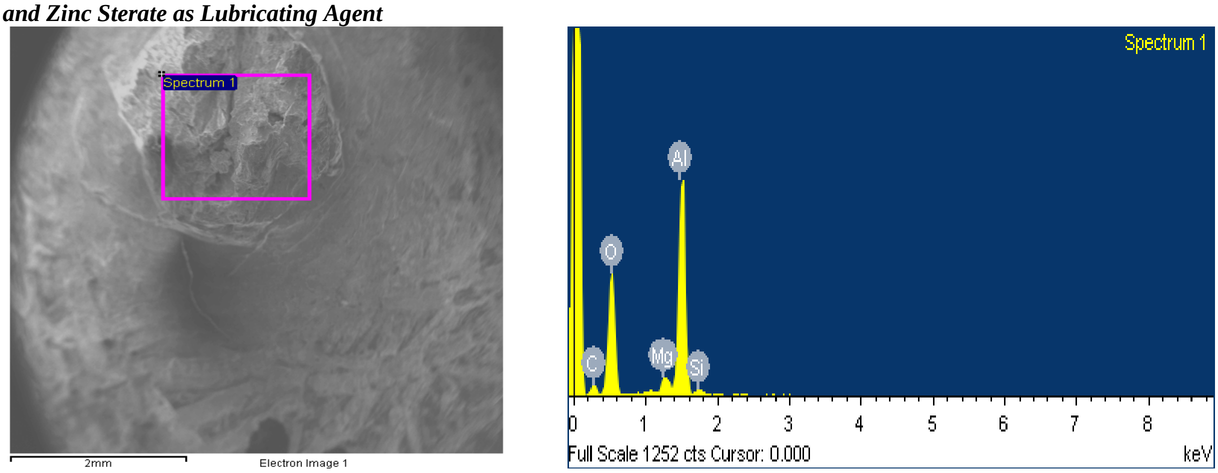


Figure 5: Shows the EDX Image of the FGM where Pure Al is mixed with 5% Weight Fraction of Alumina.

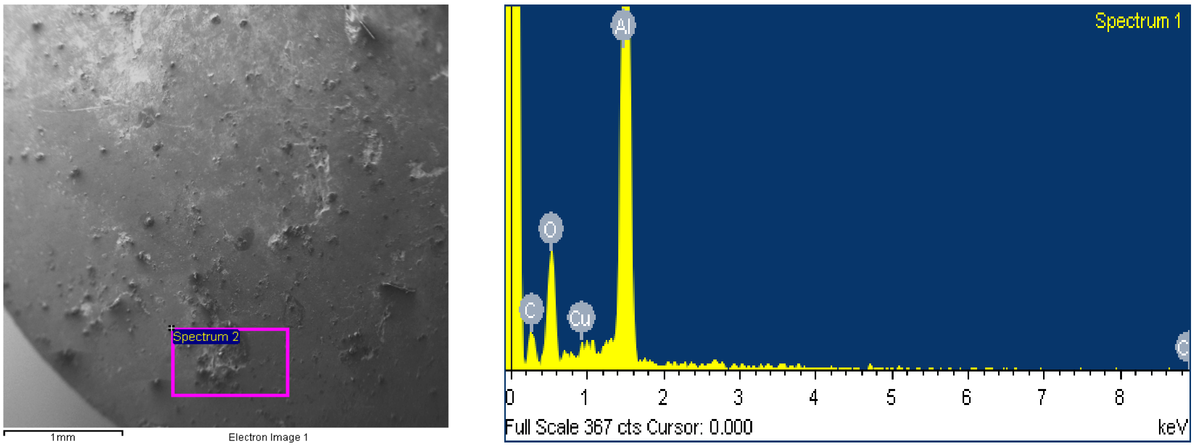


Figure 6: Shows the EDX Image of the FGM where pure Al is Reinforced with 5% of WF Copper.

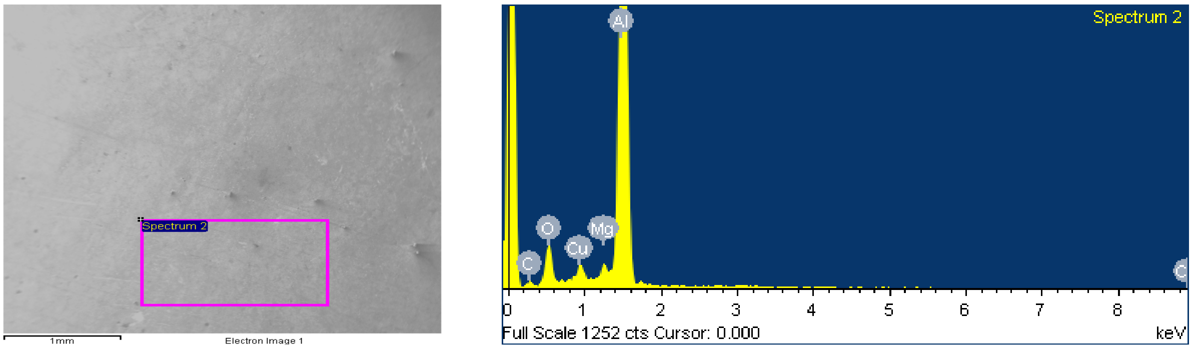
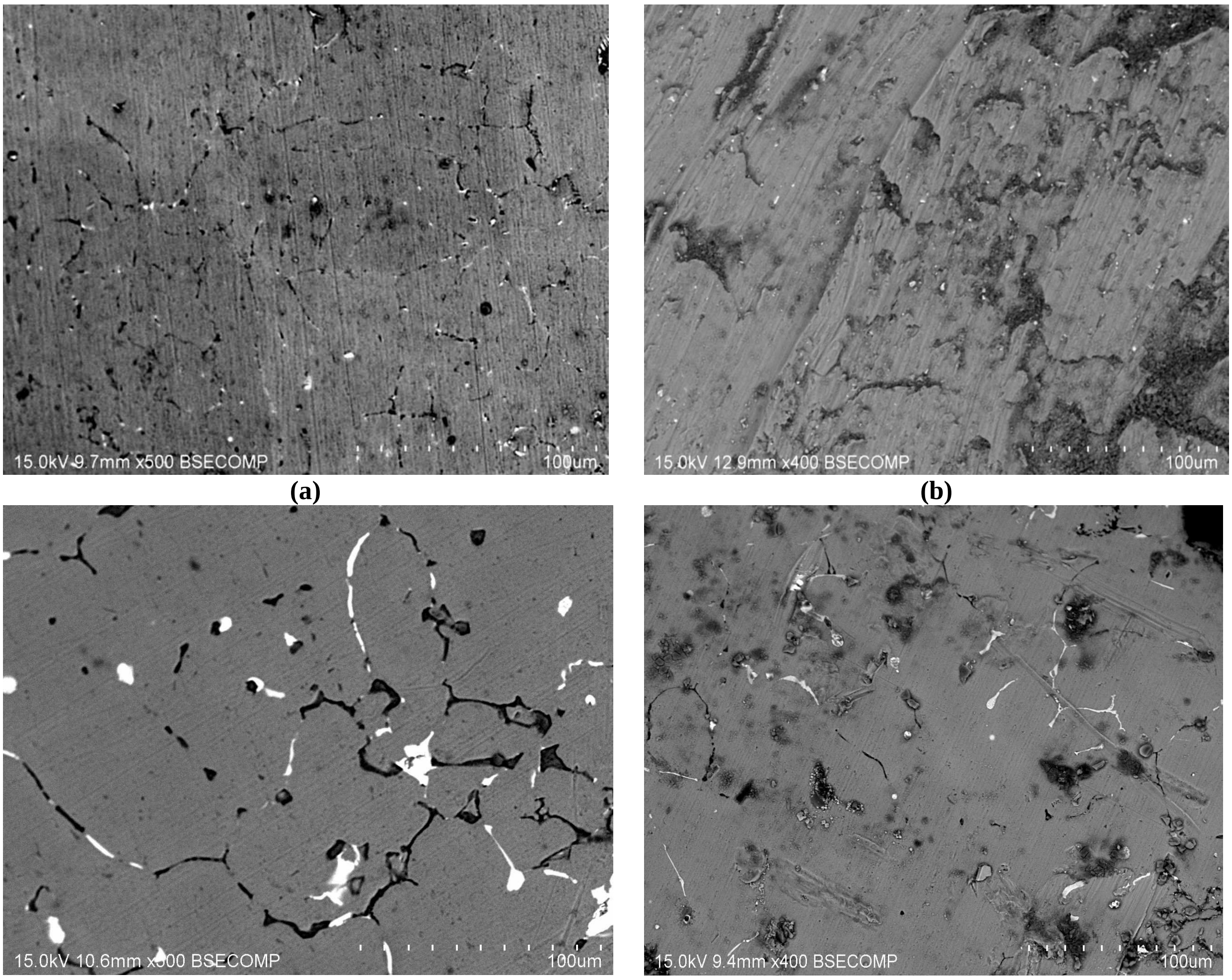


Figure 7: Shows the EDX Image of the FGM when the Copper and Alumina are Mixed in Equal Weight Proportion (5%) with Reference to the pure Al Material.

4.5. Scanning Electron Microscopic Analysis (SEM)

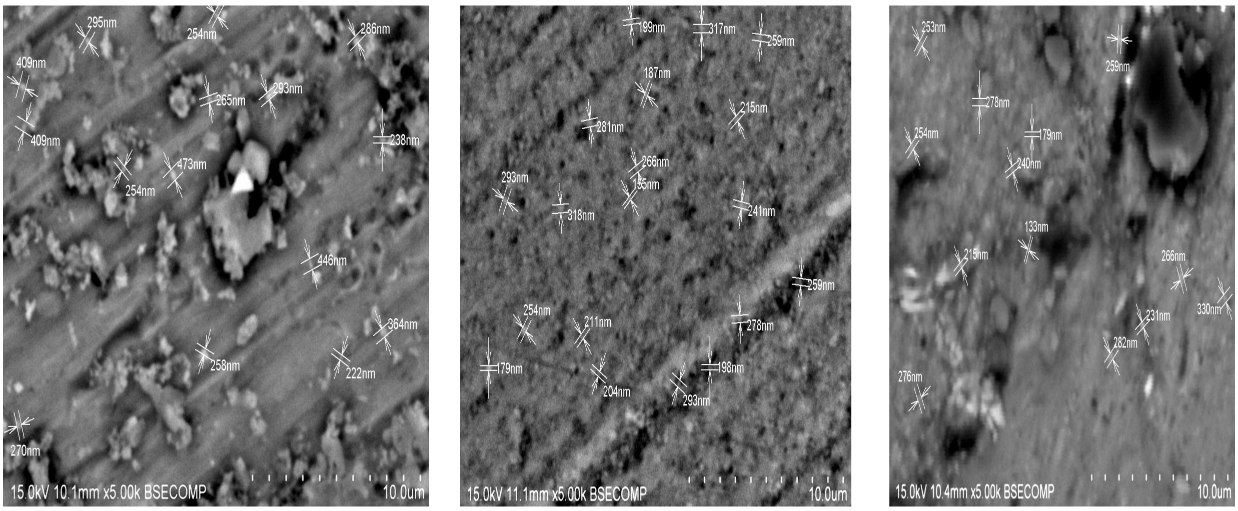
The SEM micrograph gives profuse information of reinforcement distribution, clustering, states of interface and timing details. It is observed that the functionally graded material exhibits good interfacial bonding between the reinforcement particulates and matrix material. It is also observed that there is a uniform distribution of reinforcement particulates such as alumina and copper particles in the aluminium matrix.



(a) (b)

(c) (d)

Figure 8: Represents the SEM images of (a) pure Al (b) Al with 5% Al2O3 (c) Al with 5% Cu and (d)Pure Al with 5% Al2O3 and 5% Cu.



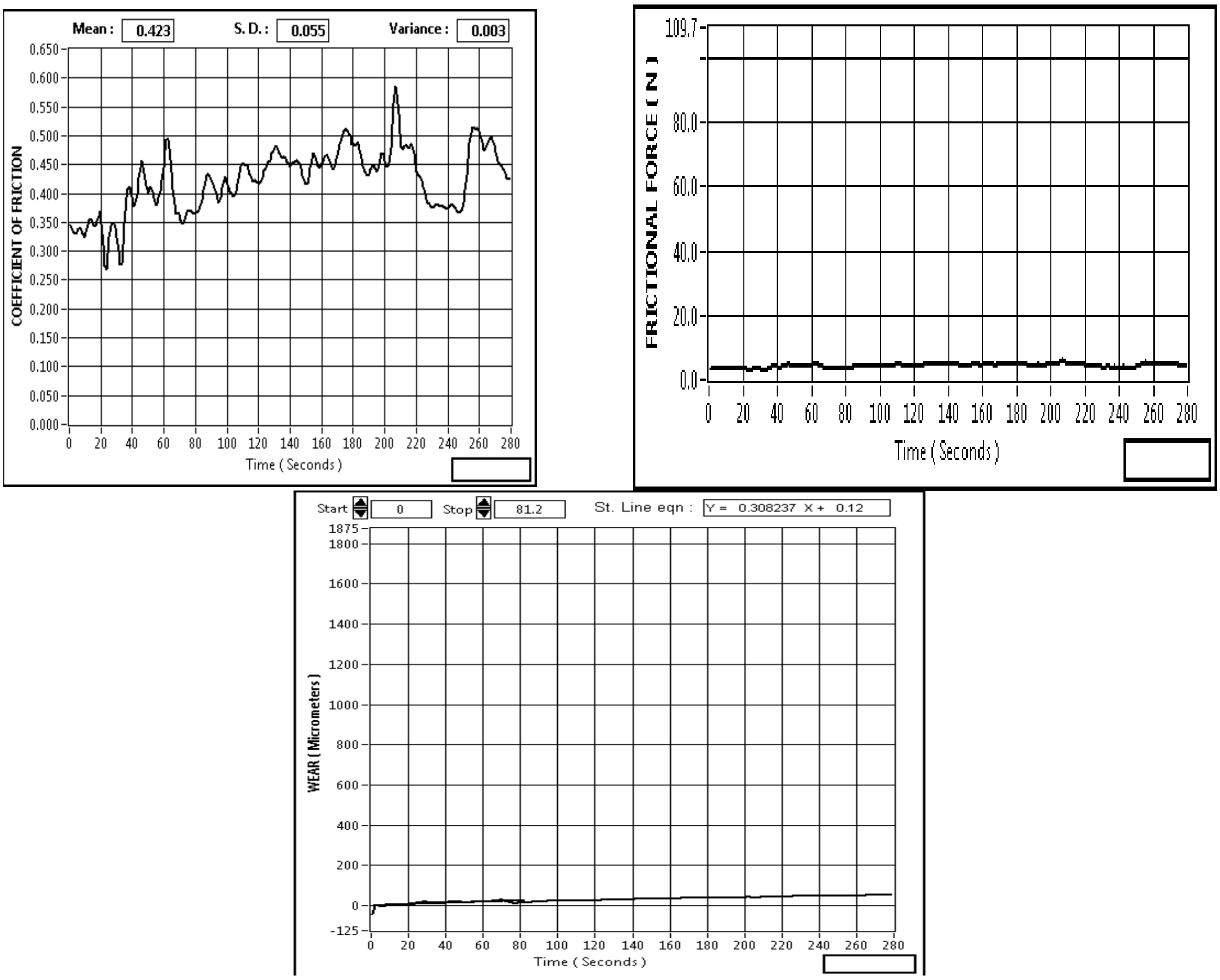
(a) (b) (c)

Figure 9: (a), (b) and (c) Shows the Particle Size Analysis of the Reinforcements, Inserting into the Void Spaces of the Parent Material.

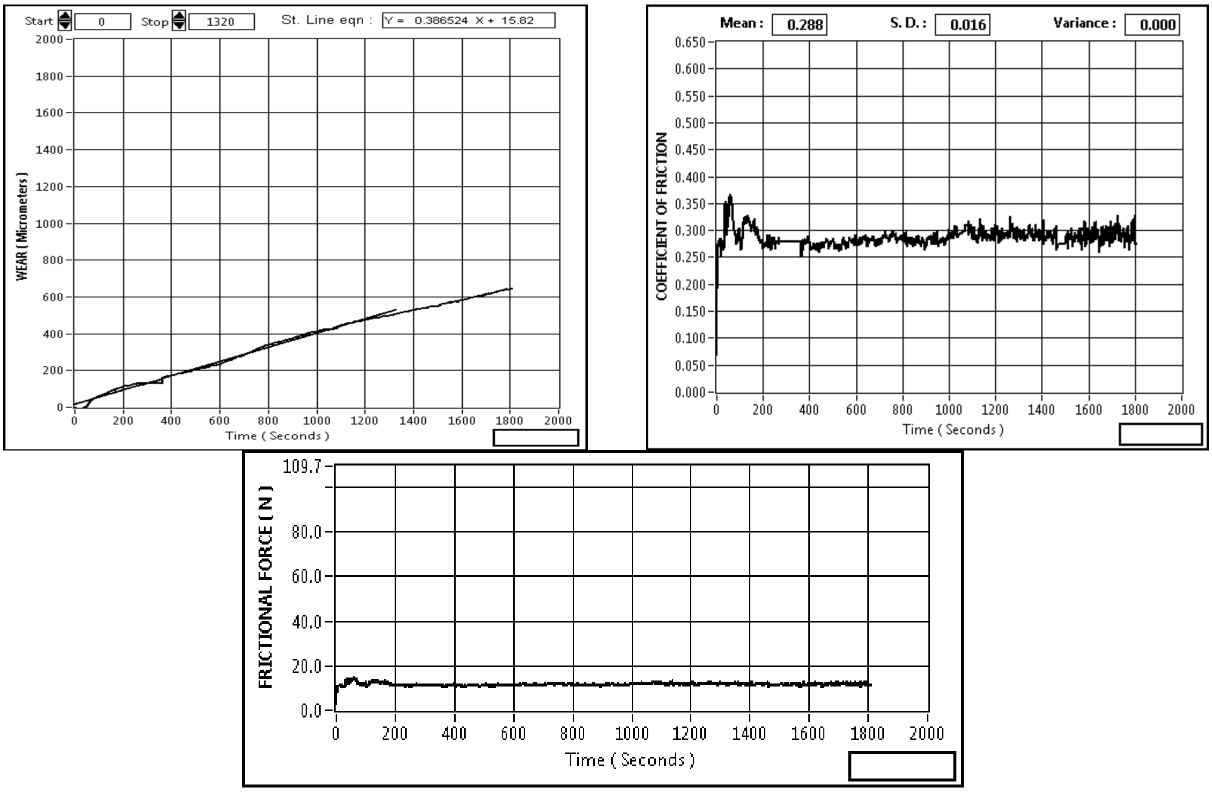
By observing the above diagrams, we can clearly visualize that the particles size and the accumulations of the particles into the vacant spaces of the matrix material. The figures (a), (b) and (c) clearly indicates this phenomenon. This phenomenon is occurring in this FGM due to the solid state processing of the composite material with the reinforcements. As the percentage of the reinforcement is increasing, the rate of agglomeration is also increased, thereby forming the clusters in the reinforcements.

4.6. Tribological Characterization

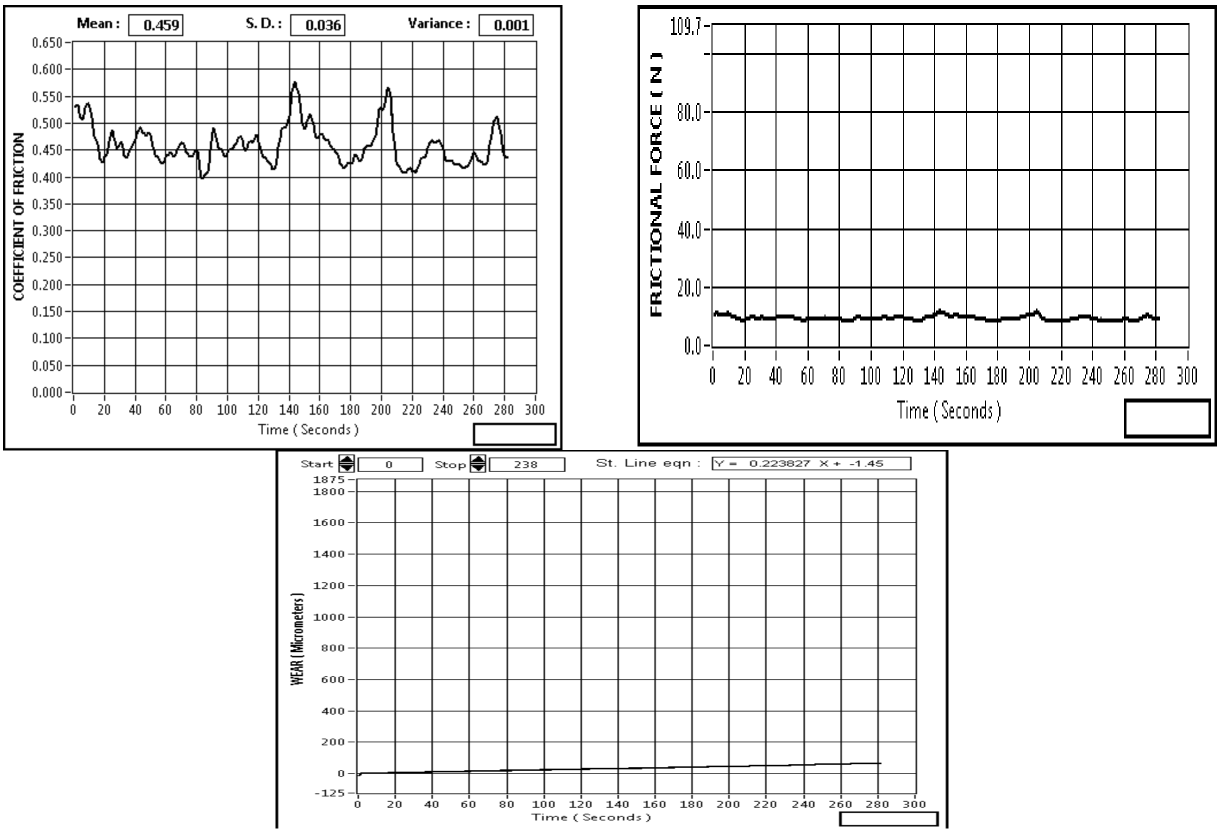
The specimens are machined, according to ASTM G33 standards and made prepared to test on pin on disc wear test apparatus of model TR-201 with 68 HRC with automatic controlling mechanism of sliding velocity, wear time and temperature. [9]



Graph 6: Showing the Coefficient of Friction, Frictional Force and Wear with Respect to Time for Final FGM (Pure Al-Alumina-Copper).

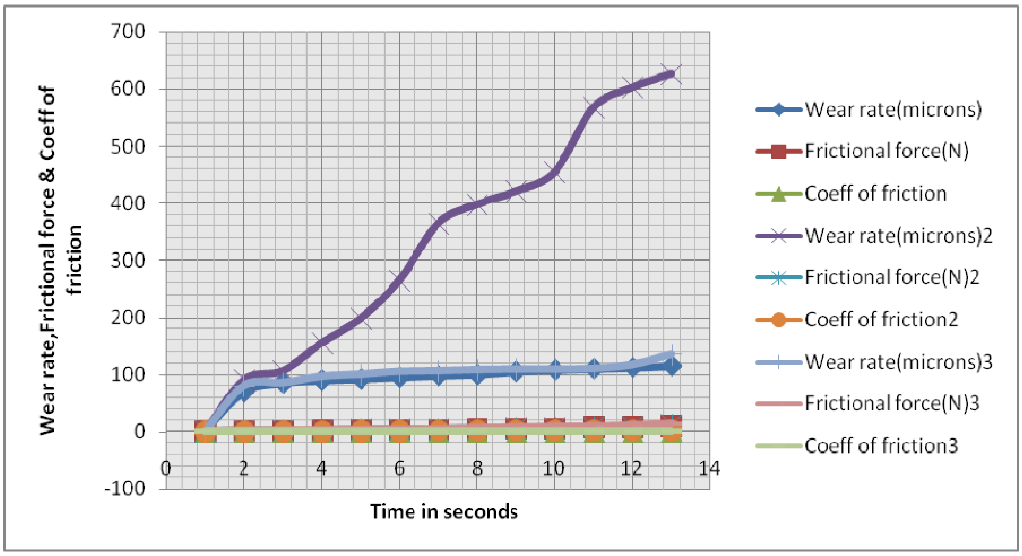


Graph 7: Showing the Coefficient of Friction, Frictional Force and Wear with Respect to Time for Pure Aluminium (98% Purity).



Graph 8: Showing the Coefficient of Friction, Frictional Force and Wear with Respect to Time for Pure Aluminium Reinforced with 10% Copper.

From the graphs 6, we can clearly evaluate that the variation of the wear rate is very low of the order of 115 microns and frictional force is 10.5N and the coefficient of friction is 0.016, having the inclusions of the alumina and the copper, but when we observe the wear graphs of the pure Al, we understood that the wear rate is of the order 627 microns, which is approximately five times more than the FGM wear rate, this is due to the findings that deposition of layer by layer material, the bonding between the matrix and the particulates is so strong enough that at higher speeds the material is not getting eroded, which we can clearly observe through SEM and EDX images(micro structural characterization). Finally, in order to assess the wear parameters, we have also synthesized FGM of pure Al and the 10% copper reinforcement and analyzed the wear curves, which are also following the same pattern followed by FGM wear results, and the values of all the characteristics is very low, which successfully shows the importance of layer wise deposition.



Graph 9: Represents the Variation of Wear Rate, Coefficient of Friction and Frictional Force when Compared with Pure Al and FGM.

5. CONCLUSIONS

After successful completion of all the experimental data, we can gather some points to make the concludatory remarks for our experimental work.

- The conventional powder metallurgy technique is replaced by automated compaction technique following ASTM G 33 standards and through this; we have successfully synthesized functionally graded materials (FGM) according to our functional requirements.
- Characterization of the material is processed in several methodologies in order to determine its mechanical, micro structural and tribological properties, which are very essential in most of the applications.
- Micro structural analysis clearly shows the deposition of materials according to layer wise, starting from 100% of pure Al as bottom layer to 80%Al+10%Alumina+10% copper on the top most layer
- Mechanical Characterization results clearly shows that the FGM properties are very high when compared to alloying materials and other unconventional materials.

- Tribological characterization clearly provides the superiority of the FGM material in terms of wear rate, coefficient of friction and frictional force when compared to pure and alloying materials.

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